

**Bond Strength and Stress Measurements in  
Thermal Barrier Coatings - 1997 Status**

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**Abstract**

To meet the aggressive Advanced Turbine System goals for efficiency, durability and the environment, it will be necessary to employ thermal barrier coatings (TBC's) on turbine airfoils and other hot section components. TBCs in production today consist of a metallic bond coat, such as an MCrAlY overlay coating or a platinum aluminide (Pt-Al) diffusion coating. During heat treatment, both these coatings form a thin, tightly adherent alumina ( $\text{Al}_2\text{O}_3$ ) film. The ceramic layer, usually 5 to 20 mils of yttria stabilized zirconia, is applied over the alumina film by plasma spray or EB-PVD processes. Failure of TBC coatings in engine service occurs by spallation of the ceramic coating at or near the bond coat to alumina or the alumina to zirconia bonds. Thus, it is proposed that the initial strength of these bonds and the stresses at the bond plane, and their changes with engine exposure, that determines coating durability.

The primary purpose of this program is to provide, for the first time, a quantitative assessment of TBC bond strength and bond plane stresses as a function of engine time and cycles, using five EB-PVD and plasma sprayed production TBCs supplied by our industrial partners. This research will generate data needed for accurate lifetime prediction models and may lead to the development of nondestructive inspection techniques for determining initial coating quality and residual life remaining for components removed from service.

The laser fluorescence technique, performed by Dr. David Clarke, UC-SB, for determining the stress in the alumina layer, shows a high compressive stress that can vary from 2 to 6 GPa in EB-PVD and plasma sprayed coatings. The stress obtained is quite consistent for specimens coated in the same batch, but varies significantly in specimens from different batches. The stress is lower for EB-PVD coatings on an MCrAlY bond coat than on a Pt-Al bond coat. The stress is also lower for plasma sprayed coatings. In preliminary tests, the bond stress in the EB-PVD/Pt-Al TBC varies in a consistent manner with thermal cycling: with the stress initially becoming more compressive, peaking, then decreasing with further cycling.

The modified ASTM direct-pull test is being used to determine the bond strength of the five coating systems. Systematic and reproducible results are being obtained, with significant differences in bond strength exhibited by different coatings. For both EB-PVD and plasma sprayed

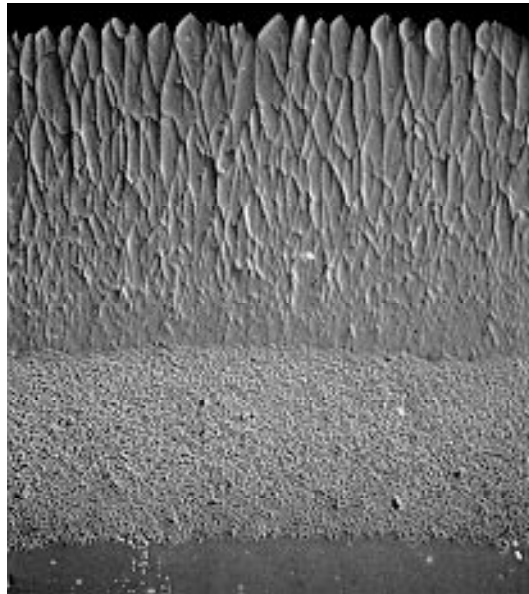
coatings, failure in this test occurs at the same location as failure occurs in service components. In preliminary tests, the bond strength in the EB-PVD/Pt-Al coating varies in a consistent manner with thermal cycling, with the bond strength initially increasing-corresponding to the increase in compressive bond stress-, peaking, and then decreasing to zero as the TBC nears spallation.

A fluorescent rhodamine dye is being used in conjunction with the direct-pull tests to clearly distinguish between fracture surface features that occur during thermal cycling and from those that occur during overload. The use of this dye is providing excellent information on damage accumulation with thermal cycling in these TBCs.

The mechanism for damage accumulation leading to spallation failure in the EB-PVD/Pt-Al coating has been determined for the first time. The Pt-Al bond coat has a coarse (100u), highly regular polygonal grain structure. It has been found that "plateaus" form above the grain boundaries on the surface of the Pt-Al bond coat by a diffusion induced grain boundary migration (DIGM) mechanism. Based on finite element analysis, these plateaus create a very high tensile stress normal to the bond coat interface. With thermal cycling, initial damage occurs as oxidation at grain boundary junctions. Damage then spreads along the grain boundaries parallel to and normal to the plane of the bond coat interface. This stress-induced oxidation and cracking of the bond coat grain boundaries eventually leads to cracking and spallation along the bond coat to alumina interface. These mechanistic observations suggest approaches for improving the spallation life of these EB-PVD/Pt-Al TBCs and these will be discussed.

*This research was performed under AGTSR Contract No. 95-01-SR030. The interest and encouragement of Dr. Daniel Fant, AGTSR Program Manager, is gratefully acknowledged.*

# **Bond Strength and Stress Measurements in Thermal Barrier Coatings**



Thermal Barrier Coating Prepared by Electron Beam Physical Vapor Deposition

**Eric Jordan, Doug Pease and Maurice Gell (Principal Investigators)**

**Jiangtian Cheng (Post-Doctoral Fellow)**

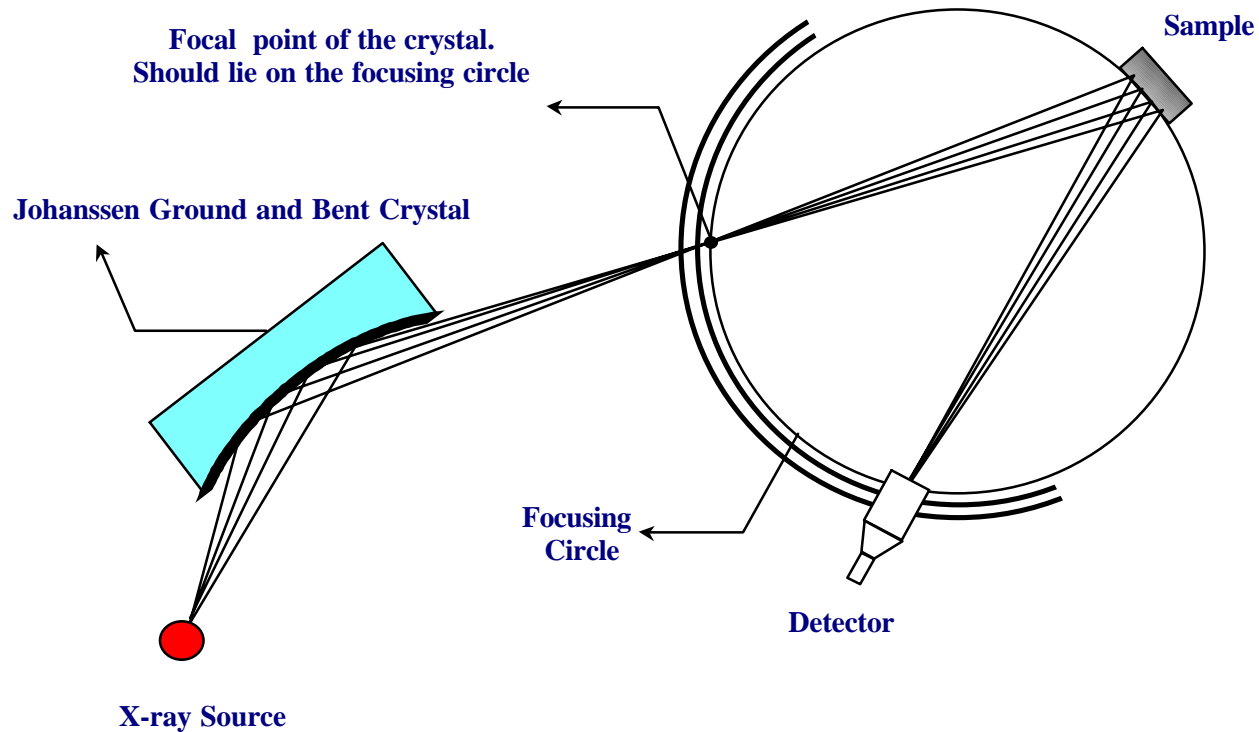
**Brent Barber , Kathleen McCarron and**

**Krishnakumar Vaidyanathan (Student Researchers)**

**School of Engineering**

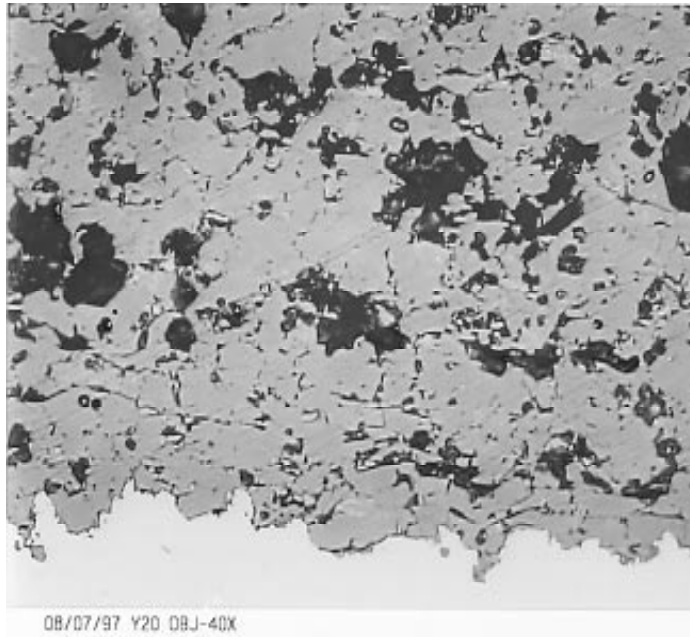
**University of Connecticut, Storrs, CT - 06269-3136**

# Enhanced X-ray Diffraction

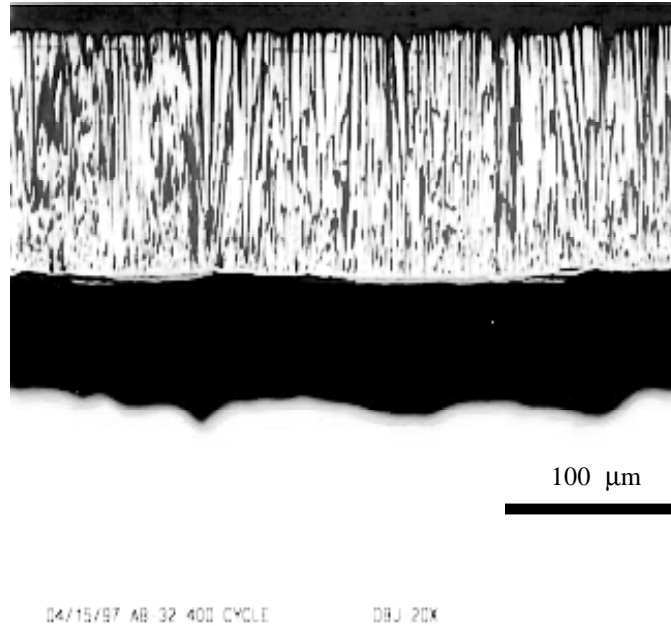


- Large bent crystal gives flux needed to penetrate the TBC.

# Rhodamine Dye Infiltrated Samples



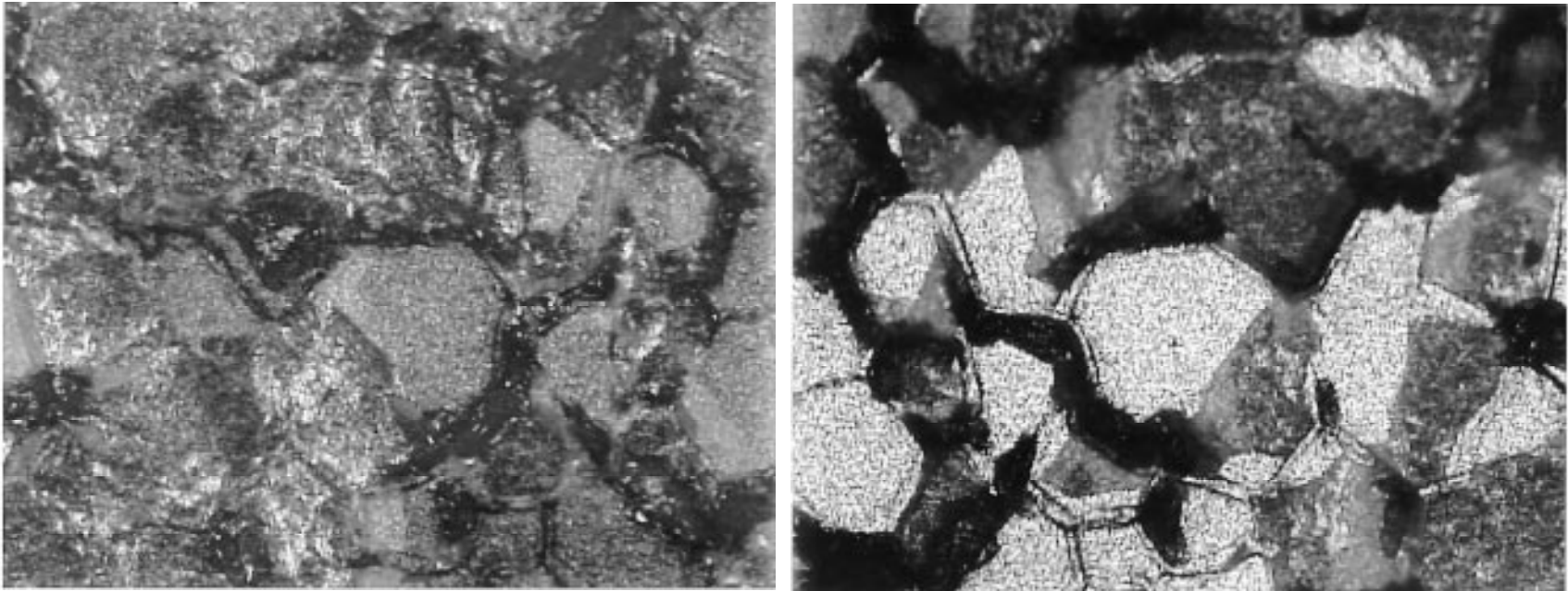
**Plasma Sprayed**



**EB-PVD**

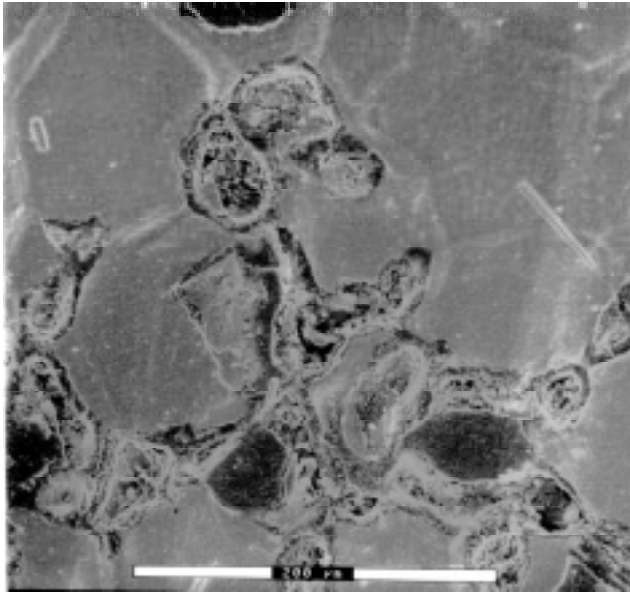
- Dye shows pre-existing defects in TBC microstructures

## Cracks and Debonds at the Interface of a Type I Specimen

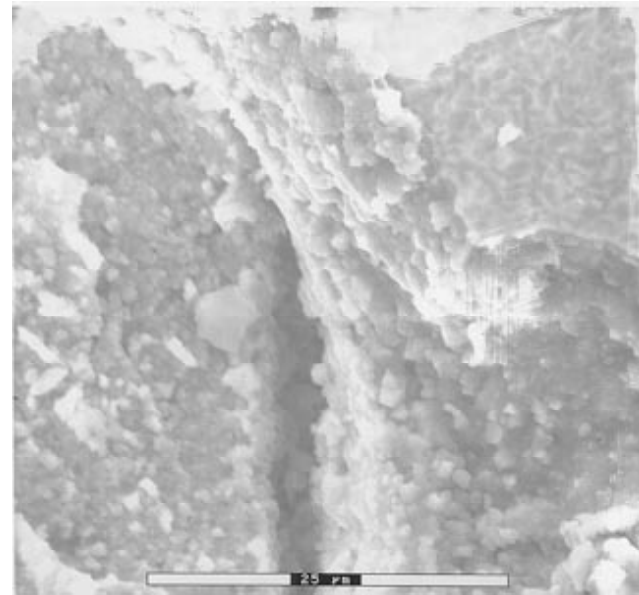


- **Fracture surface pair of a Type I uncycled sample**

# Grain Boundary Oxidation/Cracking in Pt-Al/EB-PVD Coatings

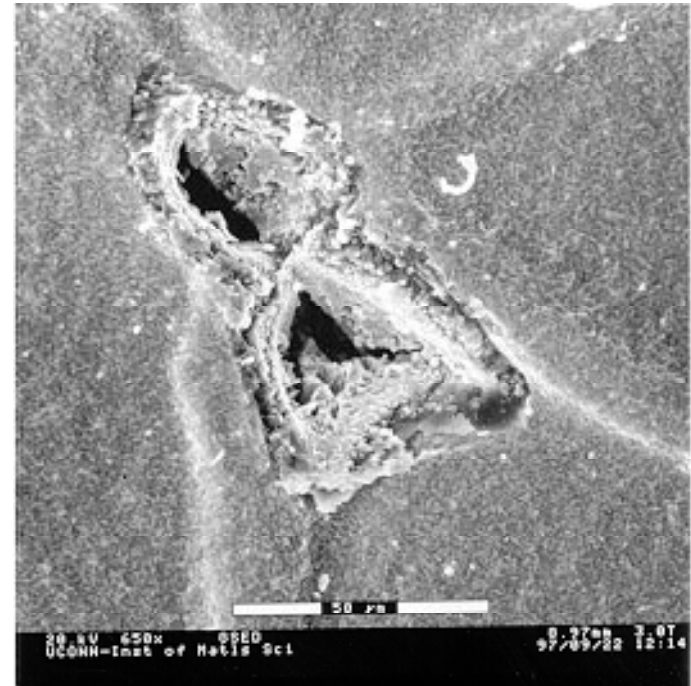
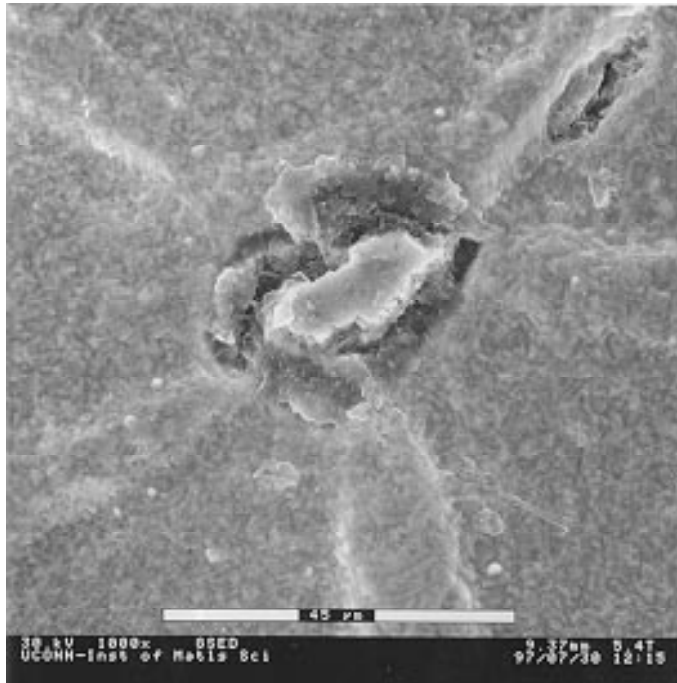


**Enhanced Oxidation along GBs  
Low Magnification**

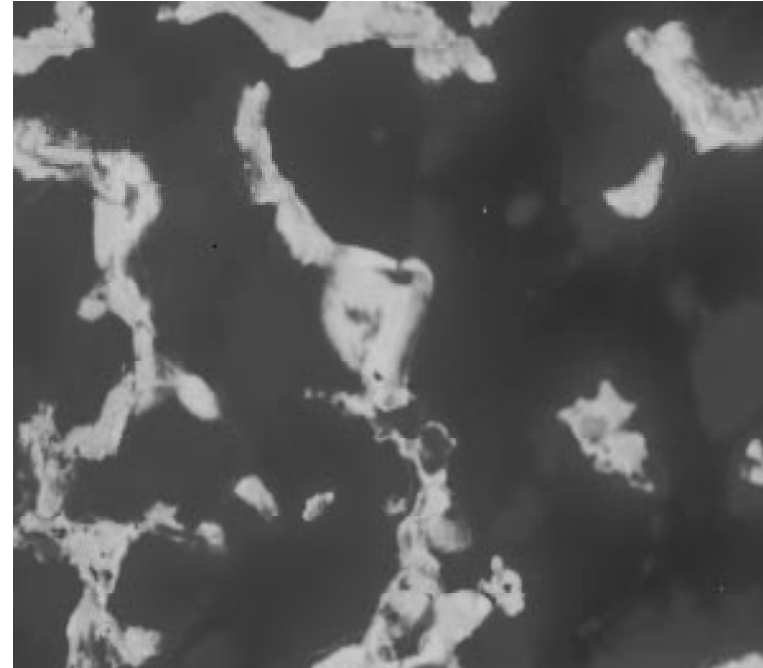
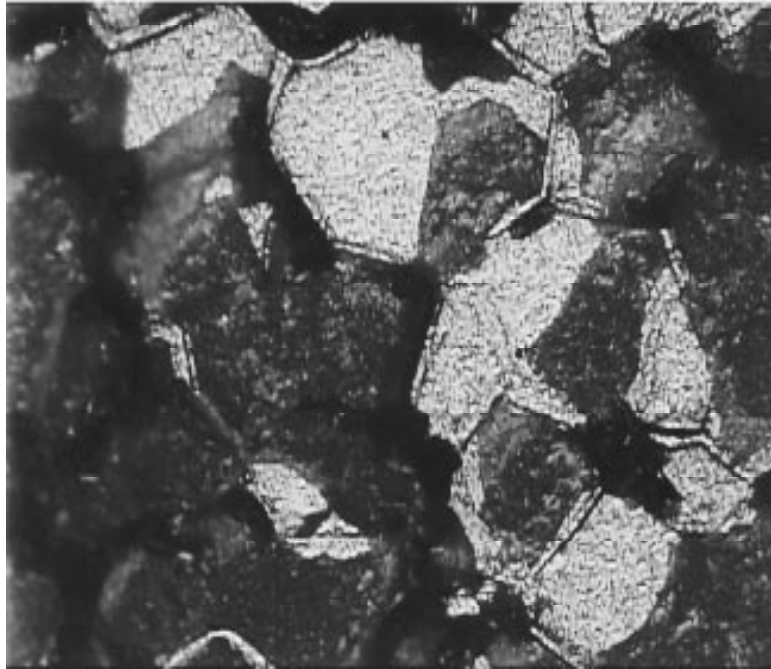


**Crack Along a GB  
High Magnification**

## Severe Oxidation at the Grain Boundary Junctions

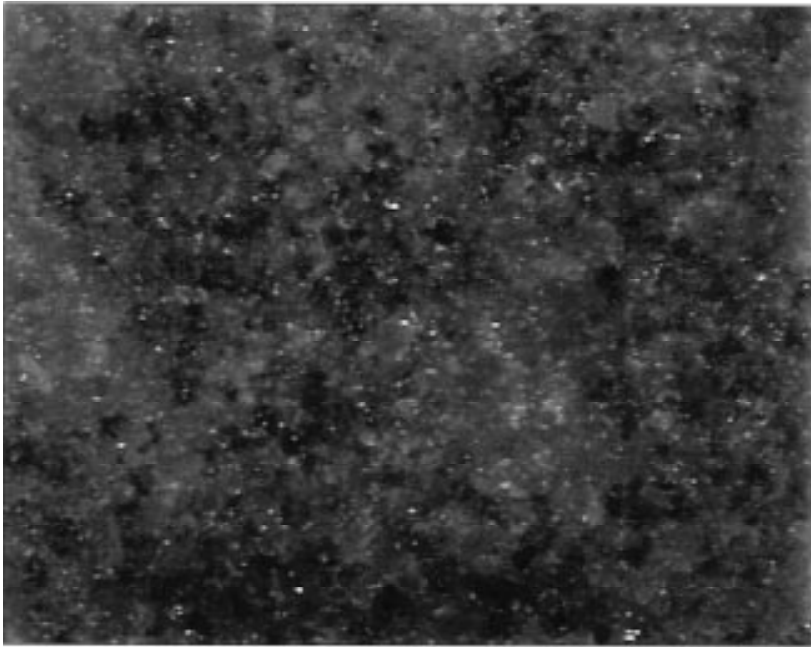


## Optical Fluorescence Shows Damage Accumulation at Grain Boundaries

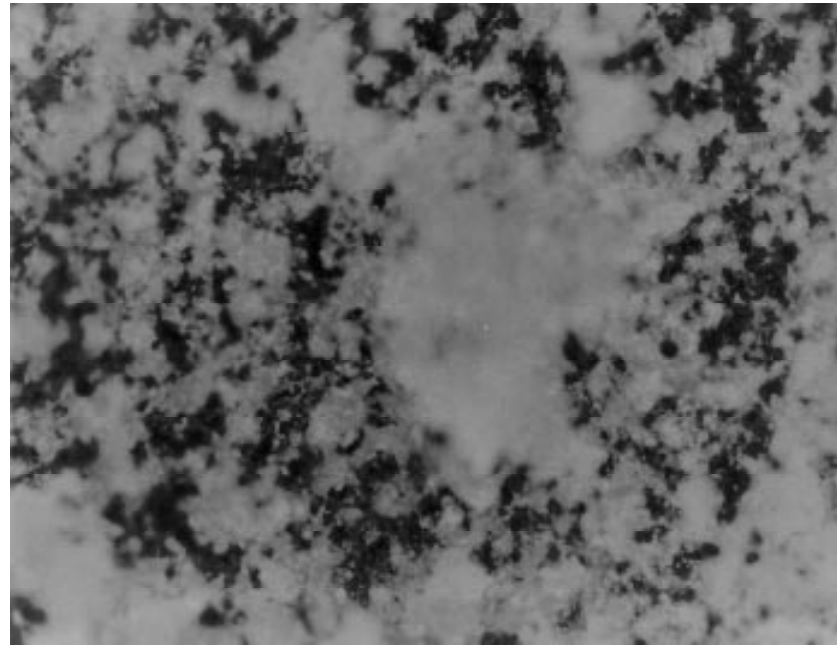


- **Comparison between optical microscope and fluorescence microscope images of Type I specimen (400-cycles).**

## **Fluorescence Microscopy Shows Incipient Damage in Plasma-Sprayed Pull Test Specimens**

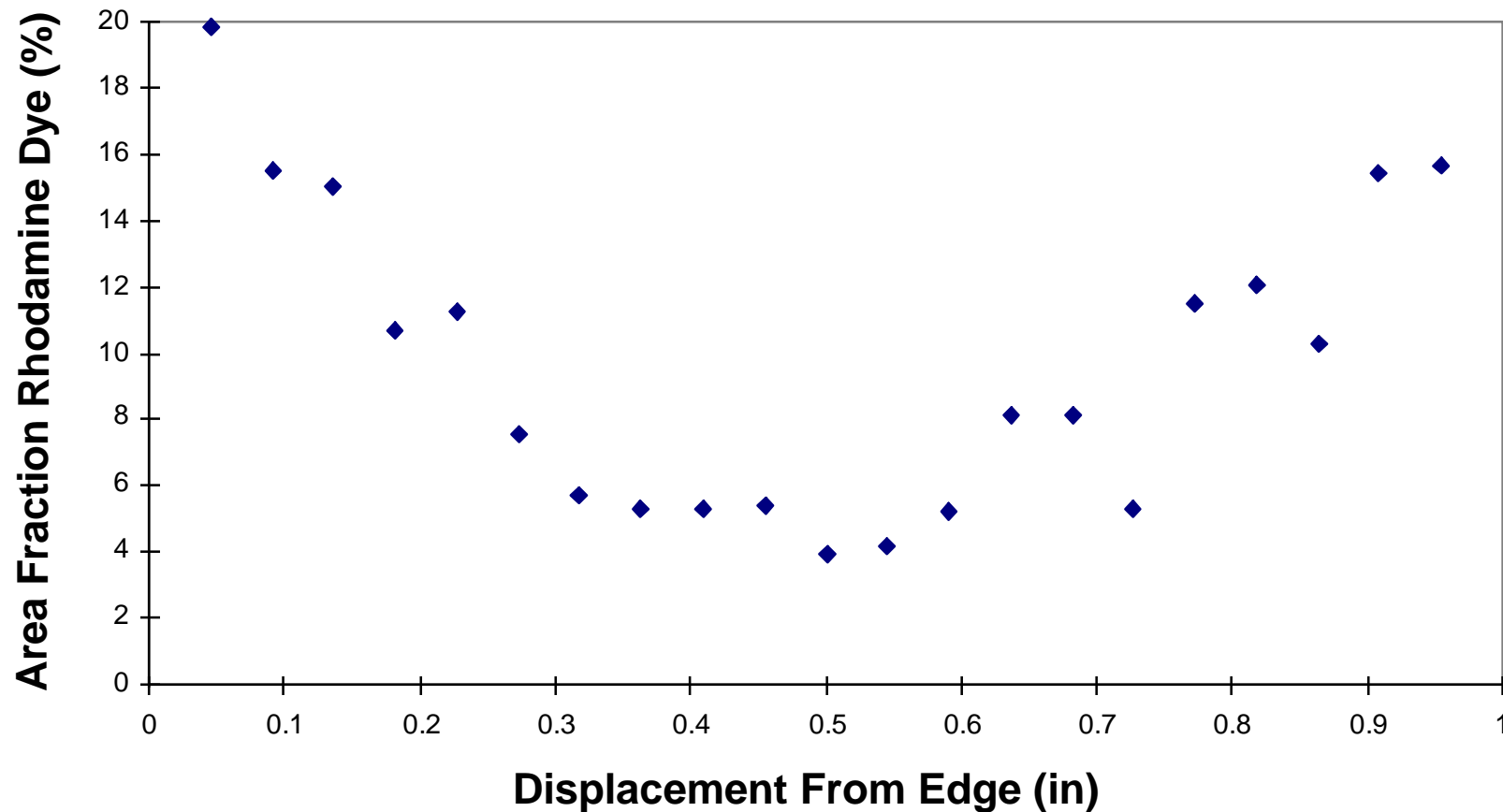


**Optical Micrograph**



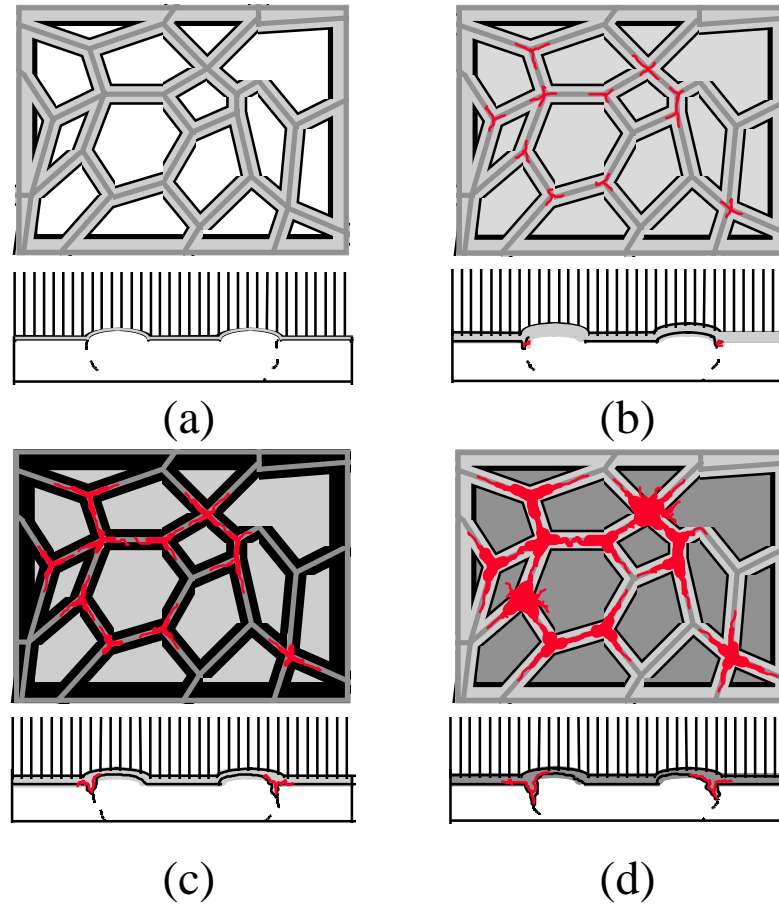
**Fluorescence Micrograph**

# Influence of Edge Effects on Incipient Damage



○ Area fraction of rhodamine dye across fracture surface suggests edge effects.

# Failure Mechanism in Pt-Al/EB-PVD System

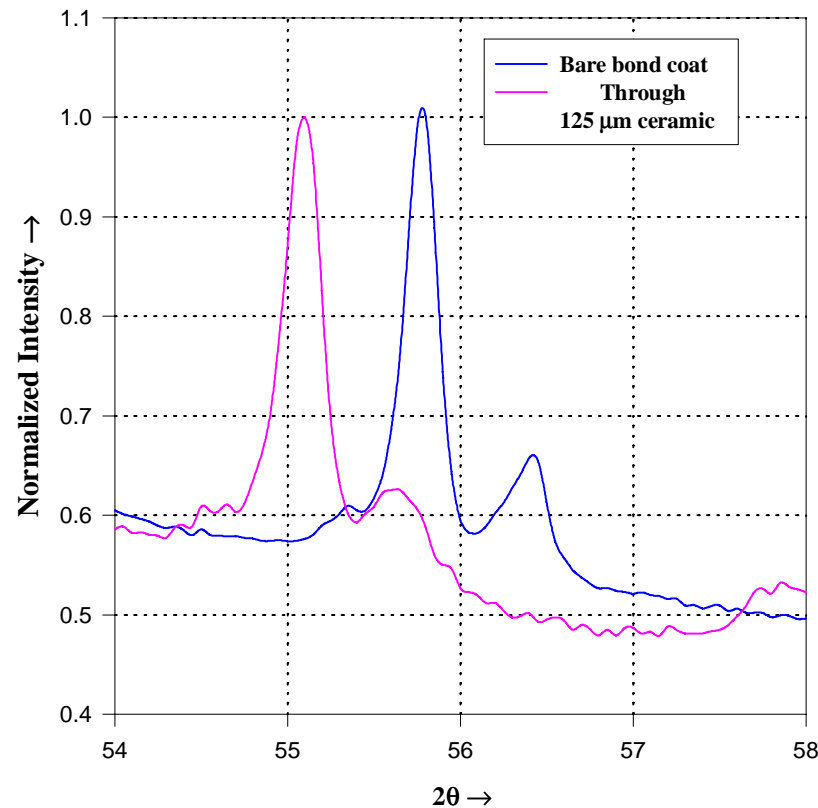


○ Schematic of Failure Accumulation with Thermal Cycles in Type I Systems

## Summary

- **Laser fluorescence, which measures stress in the thermally grown oxide, shows very reproducible results on EB-PVD TBC's as thick as 12 mils. Bond coat and processing variations are shown to influence the initial bond strength.**
- **ASTM direct pull test technique evaluated for bond strength, duplicates the service spallation location and appearance in both the plasma and EB-PVD coatings. The bond strength data generated to date shows variation among the five coatings and with thermal cycling.**
- **The laser fluorescence technique for measuring the stress in the thermally grown oxide shows great potential for becoming an NDI technique that can be used to determine initial coating quality and life remaining for service parts.**
- **A detailed study has been made for the first time of damage accumulation and spallation in the N5/Pt-Al EB-PVD TBC. It has been found that ridges are formed on the bond coat surface, by diffusion induced grain boundary migration (DIGM), prior to the application of the ceramic. In addition preferential oxidation and cracking occurs in the bond coat grain boundaries that leads to localized spallation in the grain boundary regions**

# Laboratory X-ray Diffraction Results



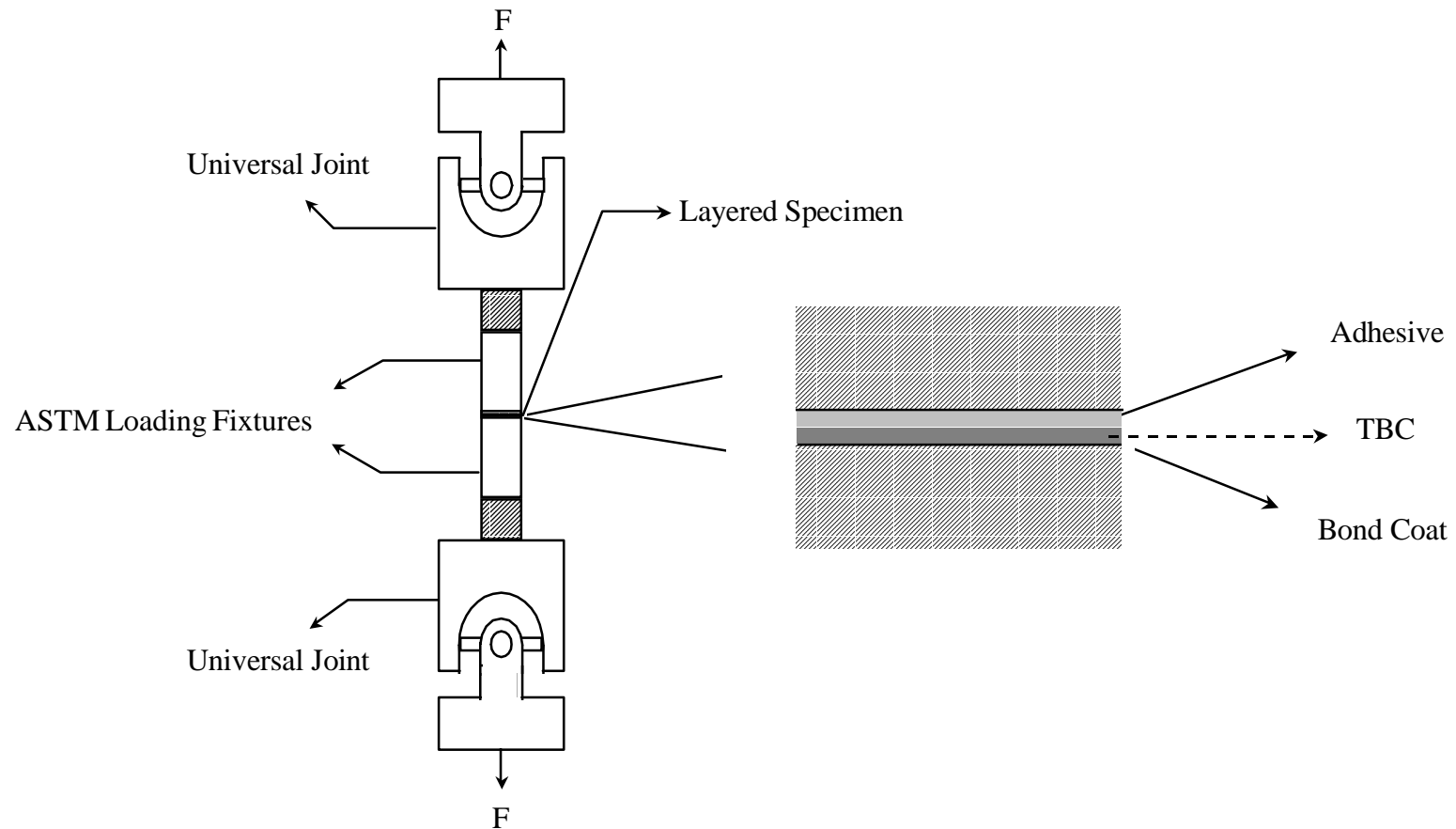
○ X-ray diffraction through a 125  $\mu\text{m}$  thick type I coating  
(Seeman - Bohlin diffractometer)

# Enhanced Laboratory X-ray Diffraction

## A Comparison of the two x-ray diffraction techniques.

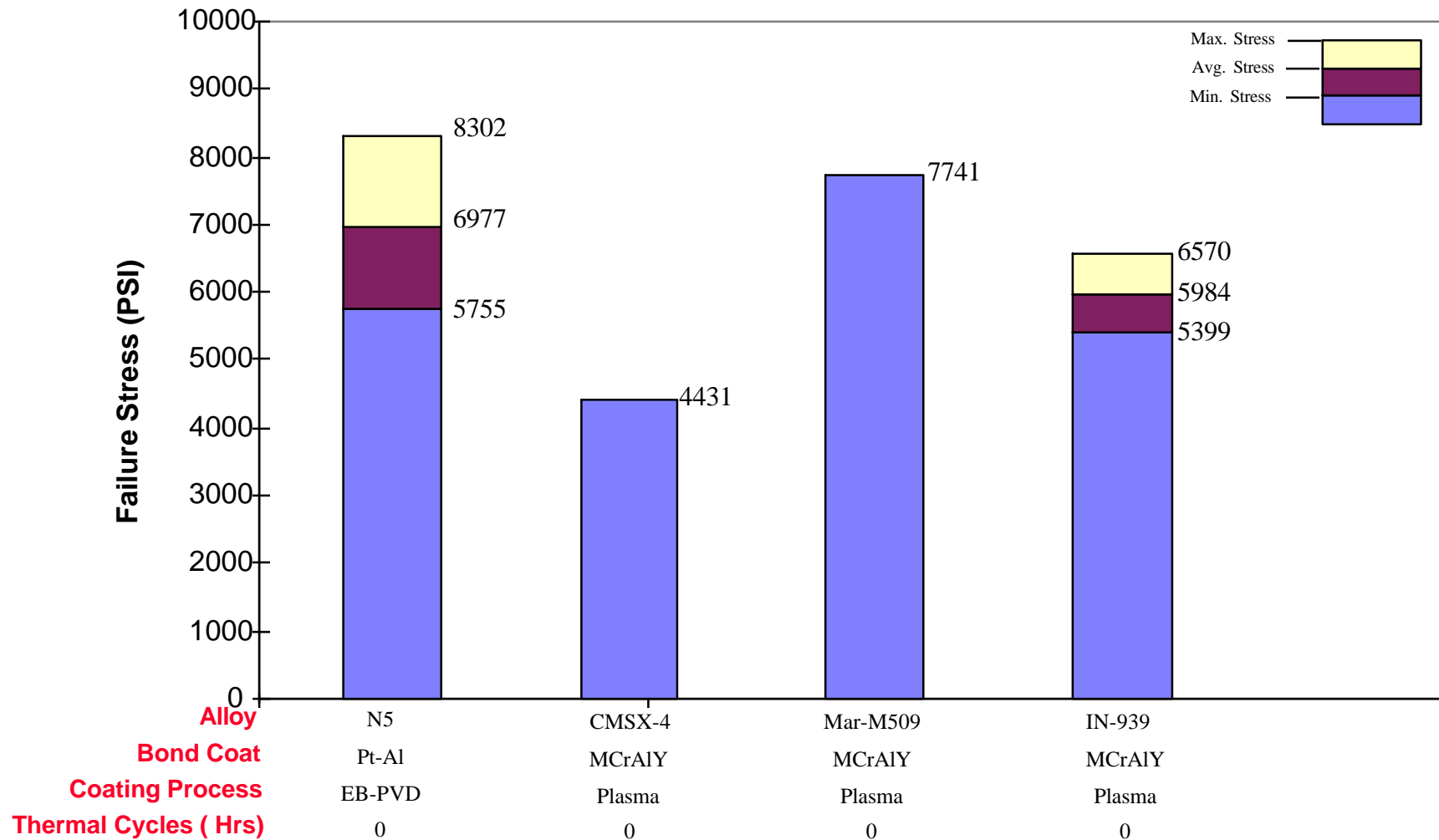
Diffraction Unit	Relative Intensity (Ratio)	Penetrating Ability (Ratio)	Detecting Efficiency (Ratio)	TBC Penetration Thickness (mils)
ORNL Diffractometer	1	1	1	0.5
Seeman-Bohlin ( Mo $K_{\alpha}$ )	10	1	1	5.0
Seeman-Bohlin ( Nb $K_{\alpha}$ )	10	10	10	10.0

# Modified ASTM Direct Pull Test



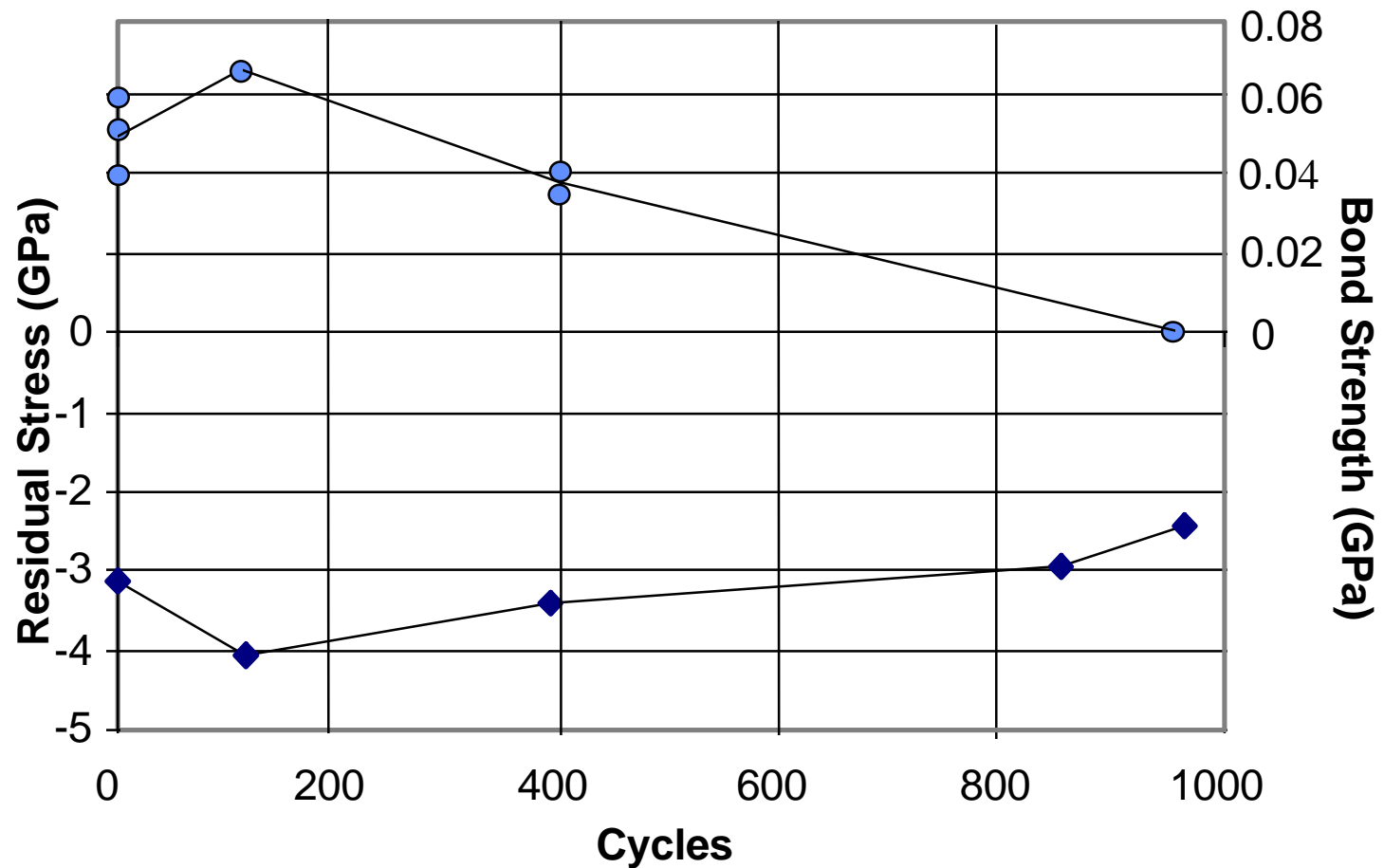
○ **Modified bond strength measurement technique.**

# Direct Pull Test Results



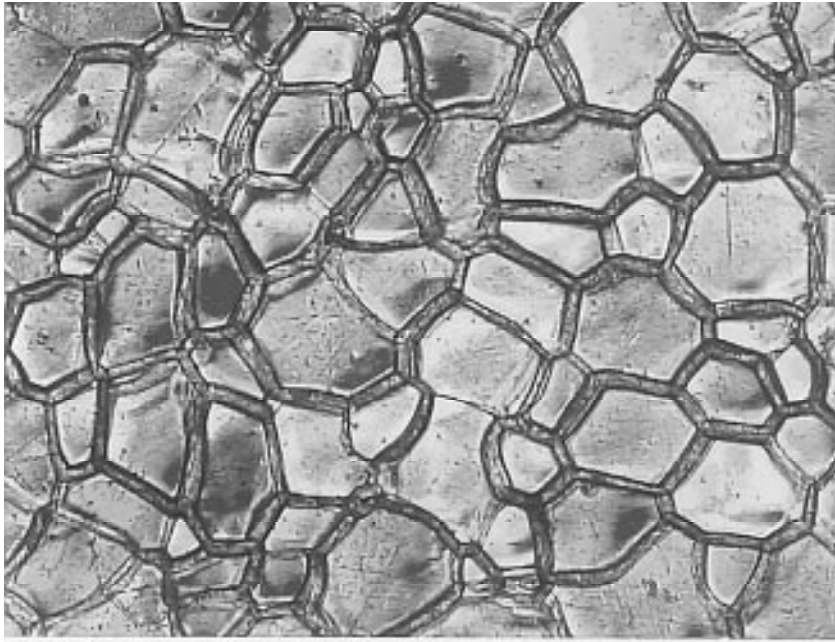
○ Bond strength varies with coating type and thermal cycles.

## Strength and Stress Comparison

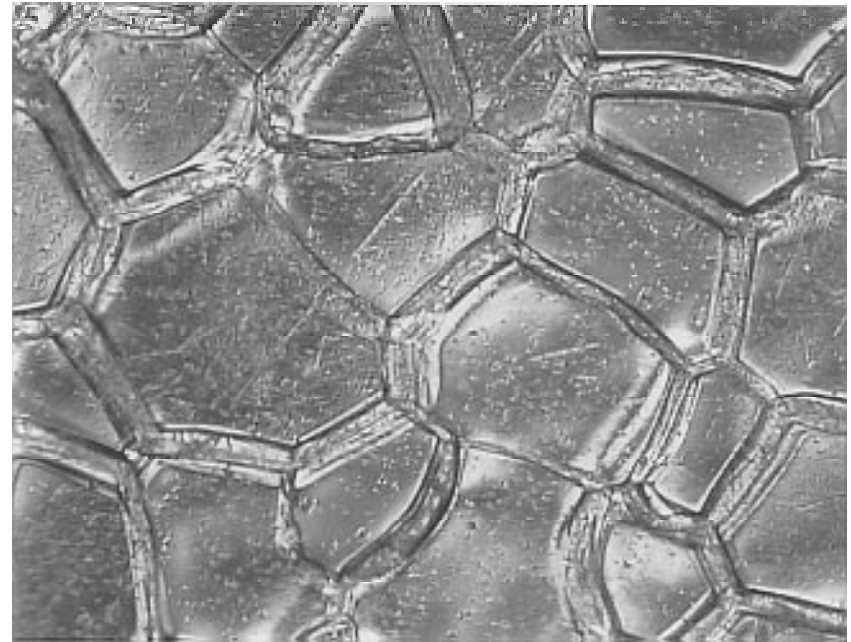


○ Residual stress and bond strength versus cycles.

## Morphology of Plateaus on Bondcoat Grain Boundaries on the Back Side

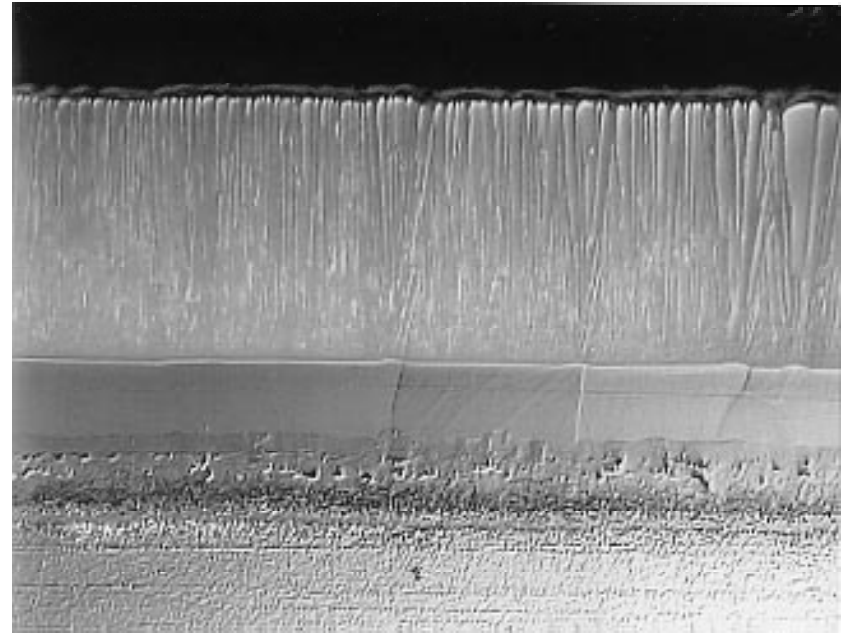
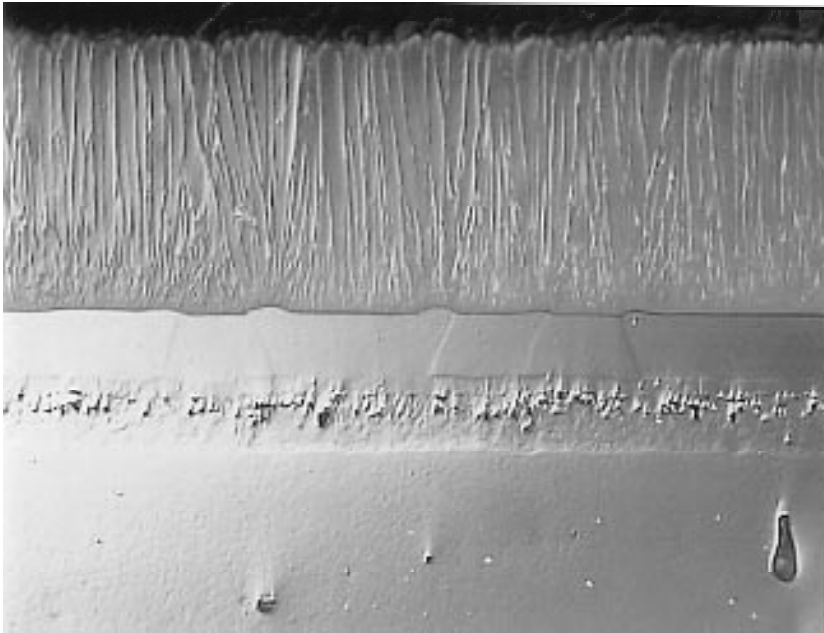


Magnification: 126×



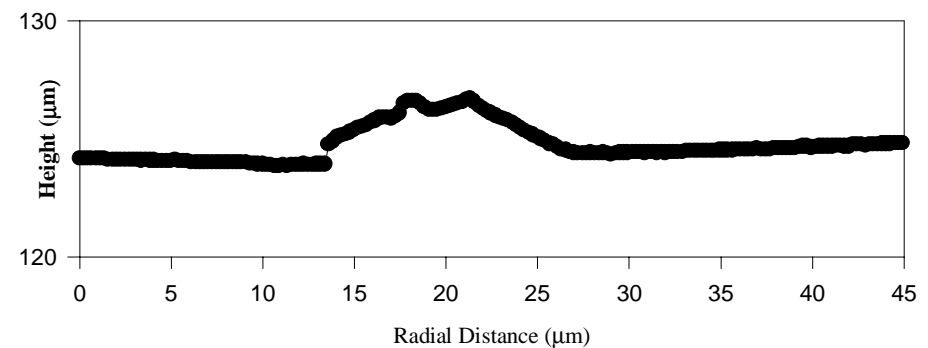
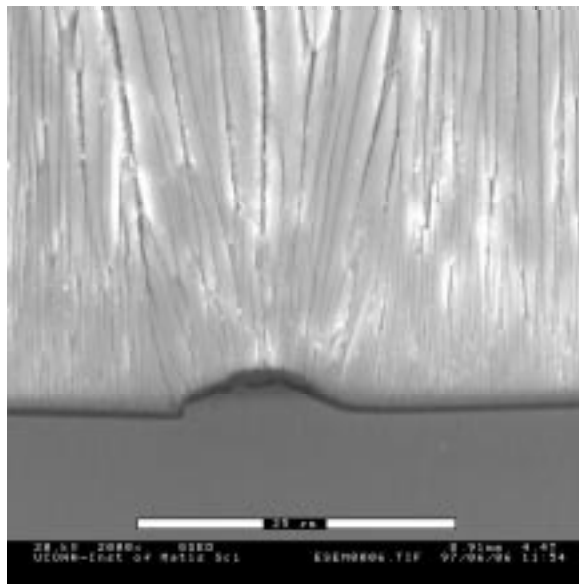
Magnification: 252×

## Association of Grain Boundaries with Plateaus

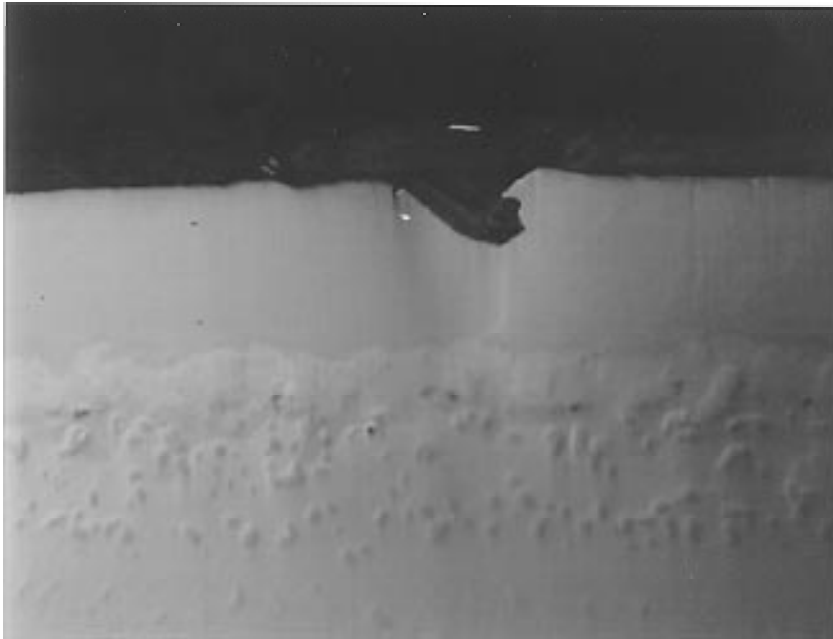


**Magnification: 252×**

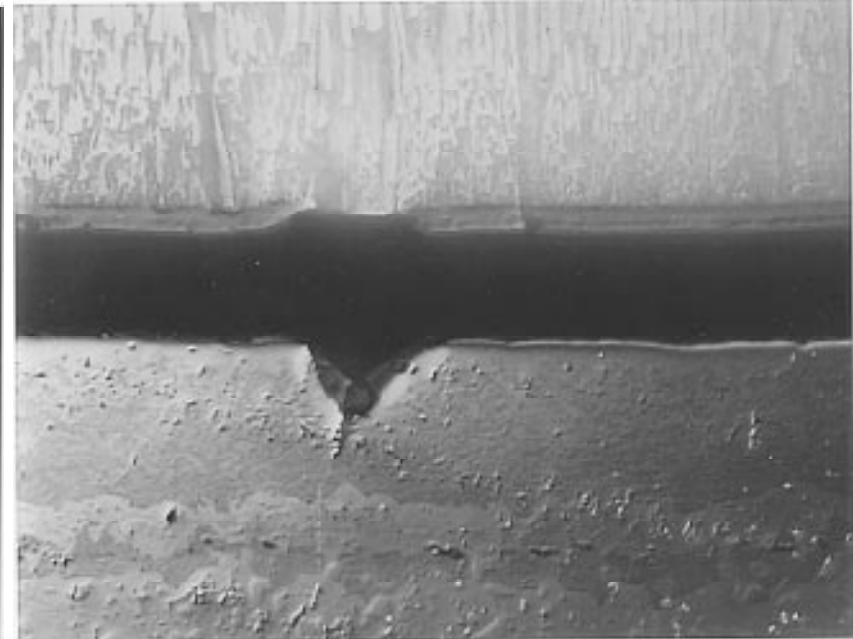
# Numerical Profile Extraction of Ridges Formed on the Grain Boundaries



## Preferential Oxidation Along the Grain Boundaries in Pt-Al/EB-PVD Coatings

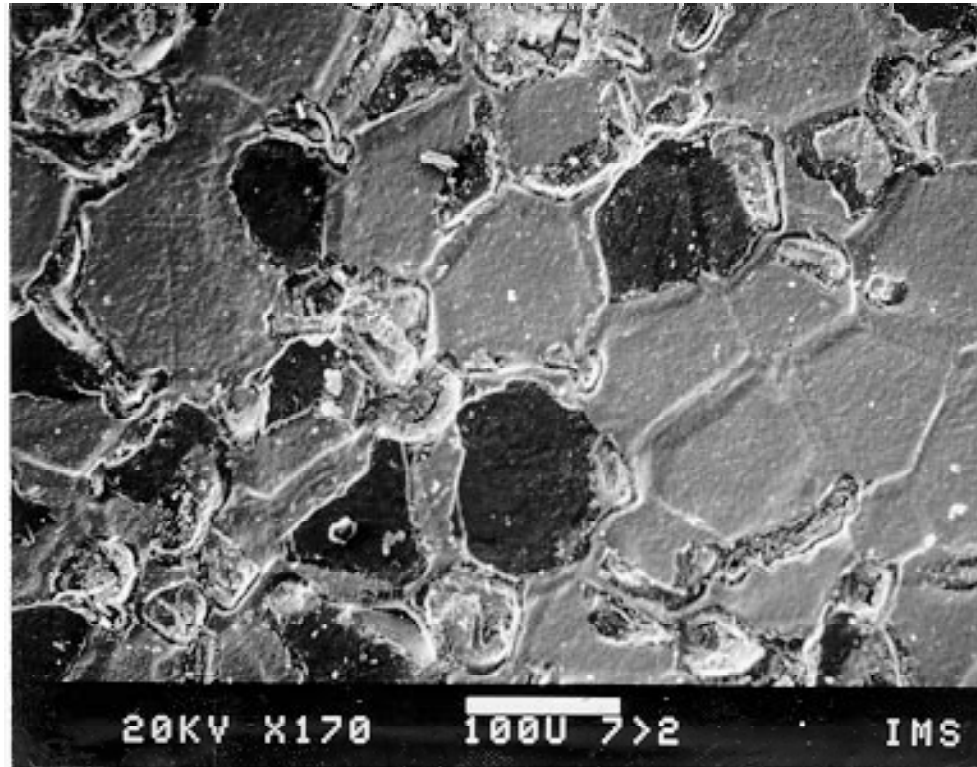


- Crevice formation along the plateaus and the grain boundaries.



- Remnant oxide scales in the interior walls of the crevices.

## Grain Boundary Oxidation/Cracking in Pt-Al/EB-PVD Coatings



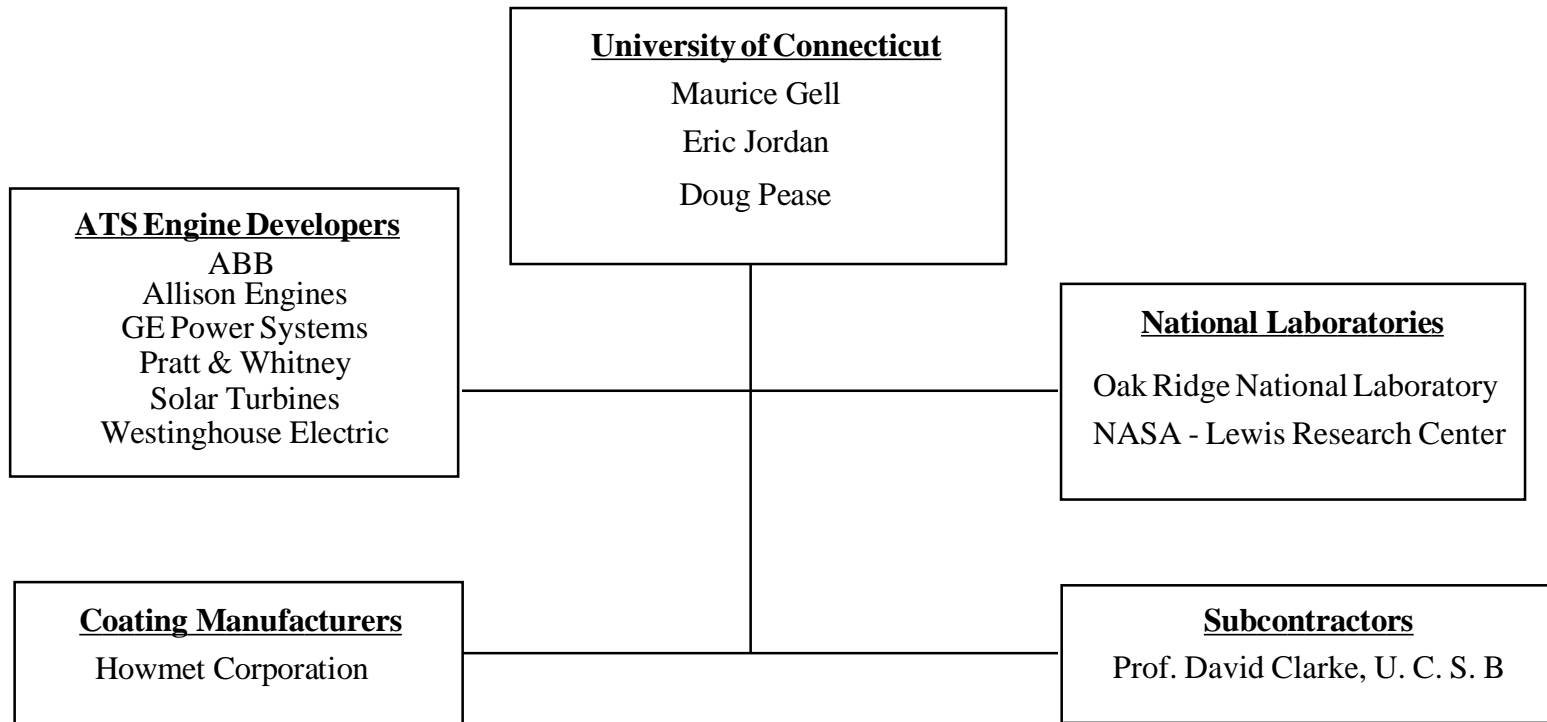
**Superalloy side ( N5 / Pt-Al / EB-PVD TBC )**

- **Dark grains encompassing grain boundaries represent  $\text{Al}_2\text{O}_3$  layer**

# Program Objectives

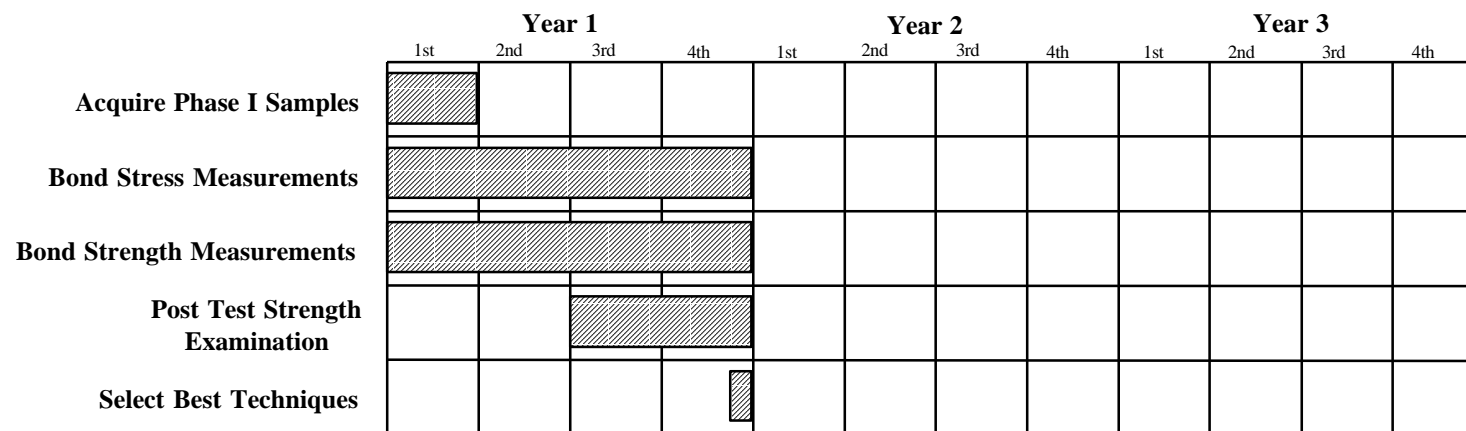
- **Determine Role of Bond Strength and Stress in Controlling TBC Life**
- **Describe Fracture Mechanisms**
- **Provide Quantitative Data for Use in Lifetime Prediction Models**
- **Develop TBC NDI Techniques for Determining Initial Coating Quality and Life Remaining**

# Project Organization

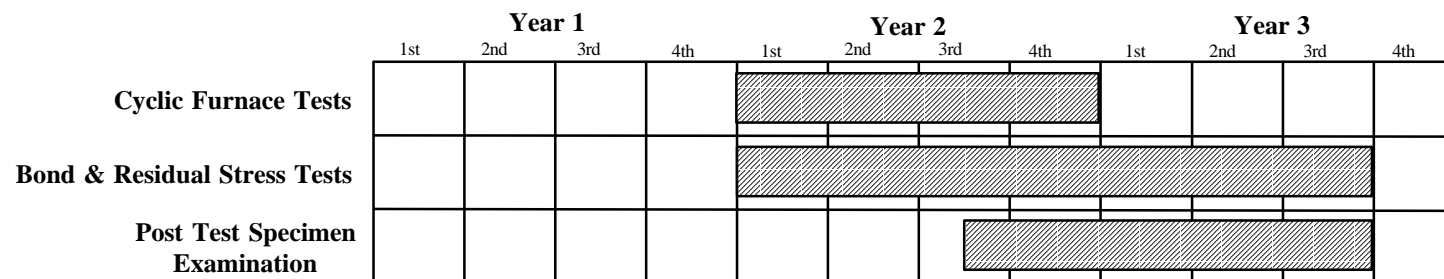


# Program Schedule

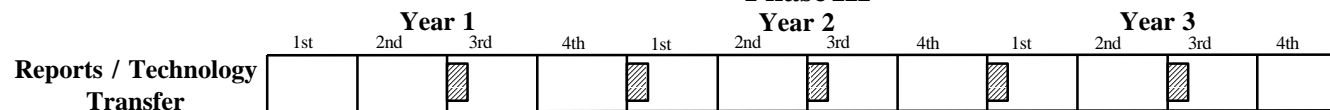
## Phase I



## Phase II



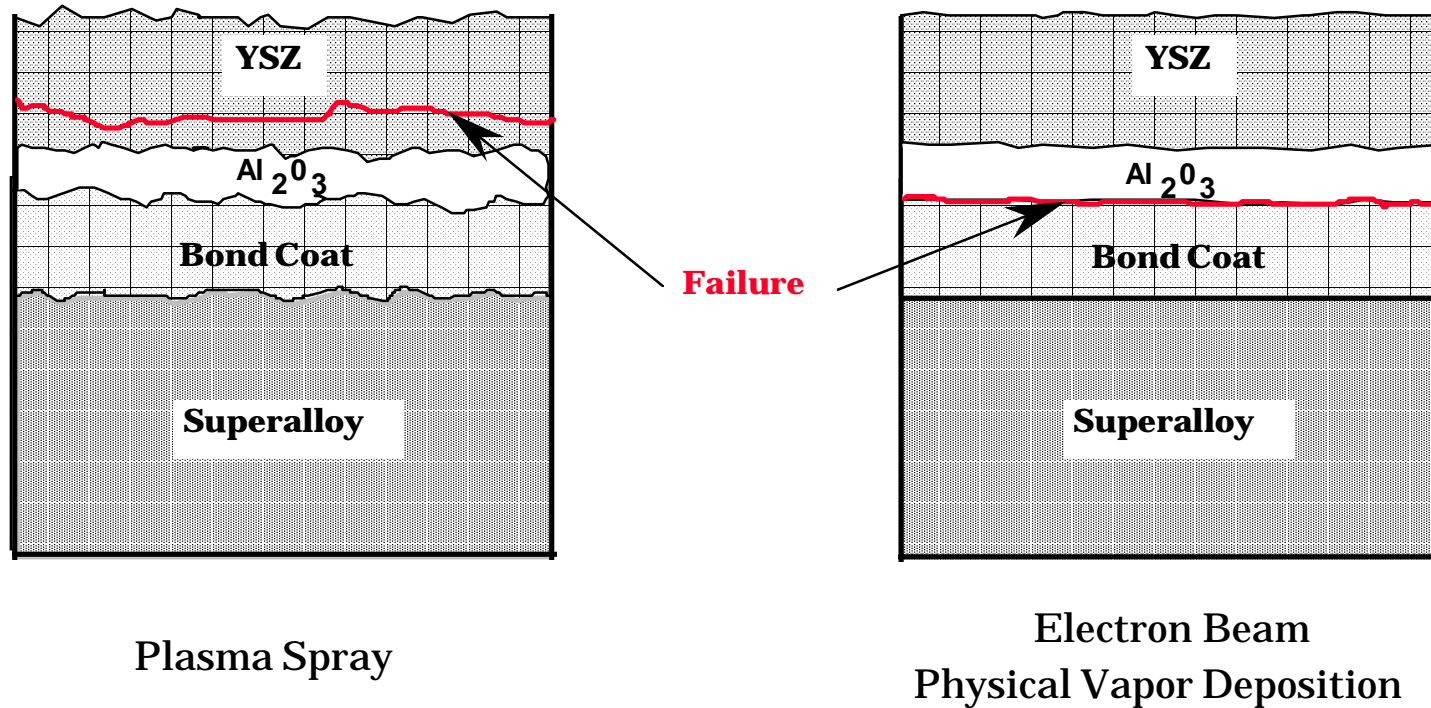
## Phase III



## TBCs Evaluated in this Program

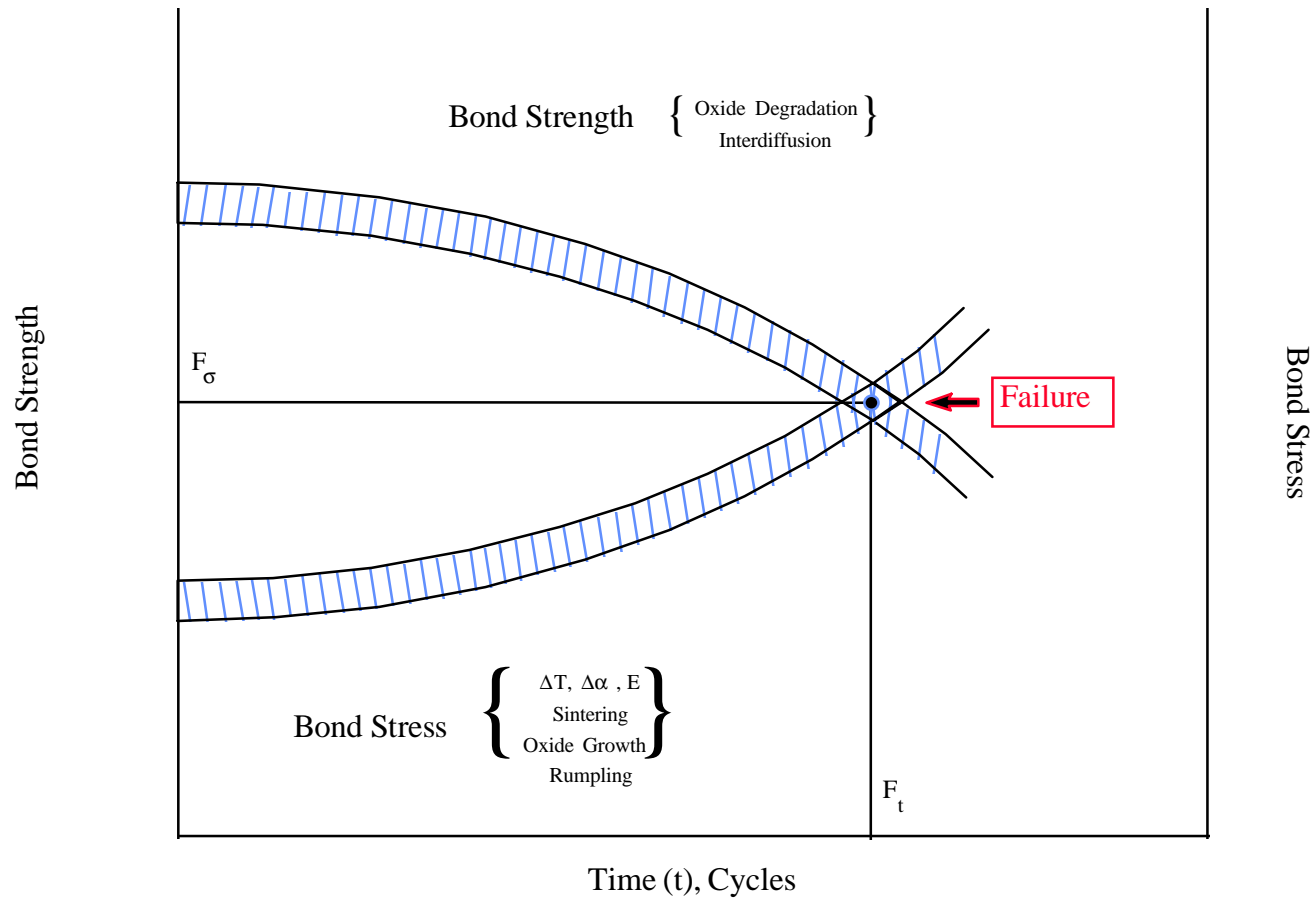
Type	Superalloy	Casting Form / Alloy Type	Bondcoat		Ceramic	
			Type	Thickness ( $\mu\text{m}$ )	Type	Thickness ( $\mu\text{m}$ )
I	N5	Single Crystal Ni	Pt - Al	65	EB-PVD	115
II	IN - 939	Polycrystal Ni	MCrAlY	70	Plasma	255
III	CMSX - 4	Single Crystal Ni	MCrAlY	120	EB-PVD	315
IV	CMSX - 4	Single Crystal Ni	MCrAlY	120	Plasma	250
V	MAR-M-509	Polycrystal Co	MCrAlY	125	Plasma	300

# Coating Failure Locations



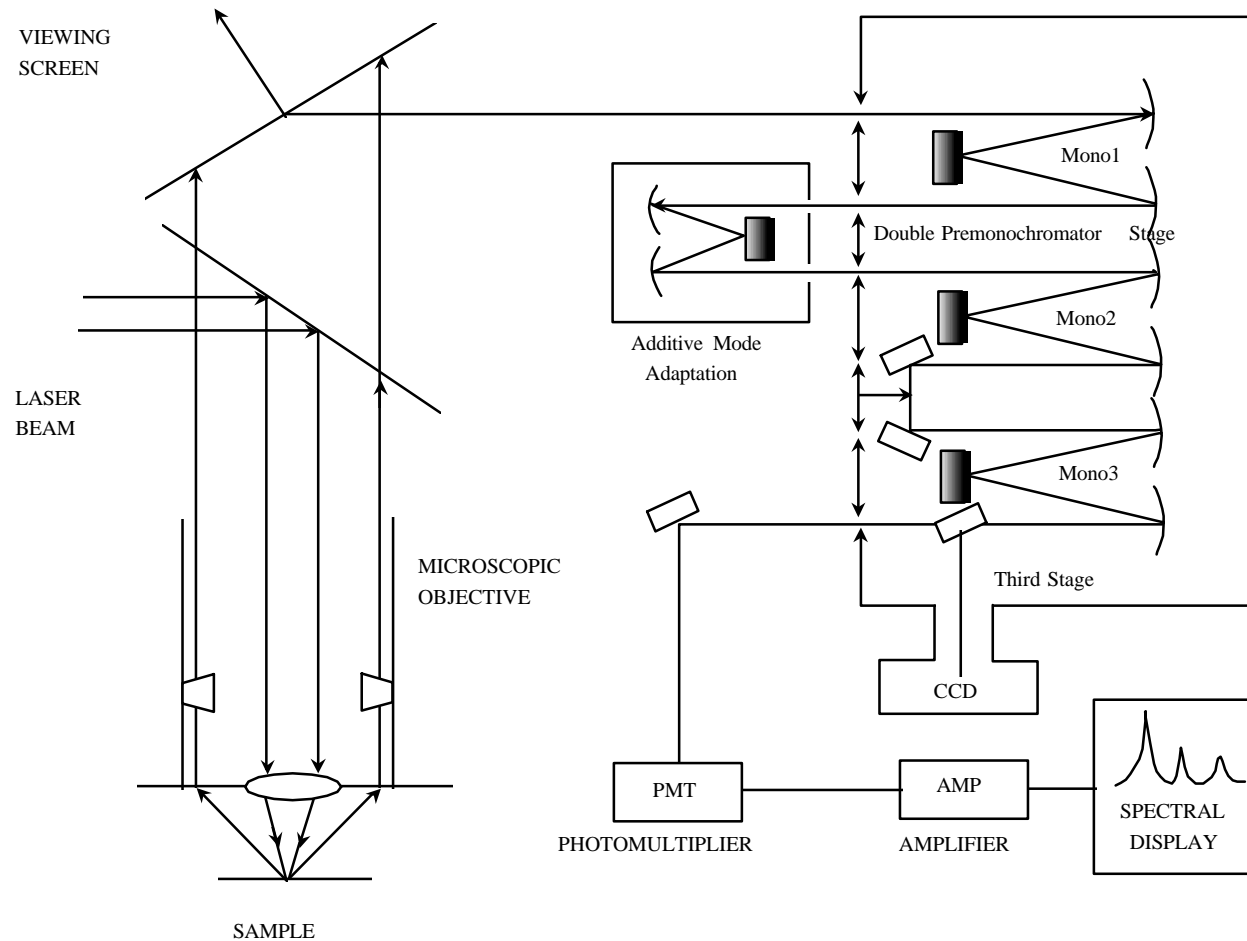
- Failure occurs predominantly at or near the ceramic-bond coat interface

# Proposed Failure Model



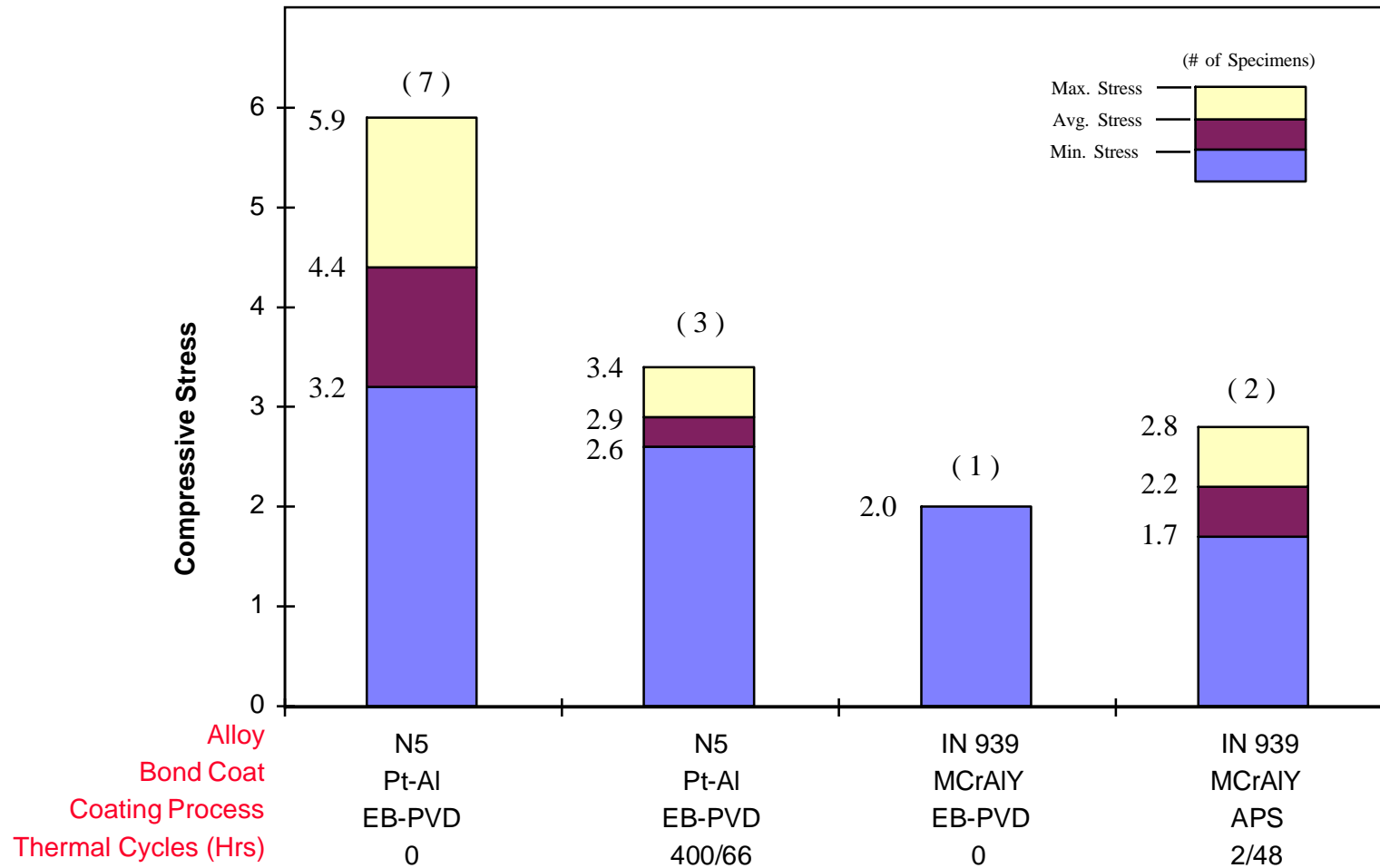
- **Failure occurs when bond plane stress equals the bond strength.**

# Laser Fluorescence



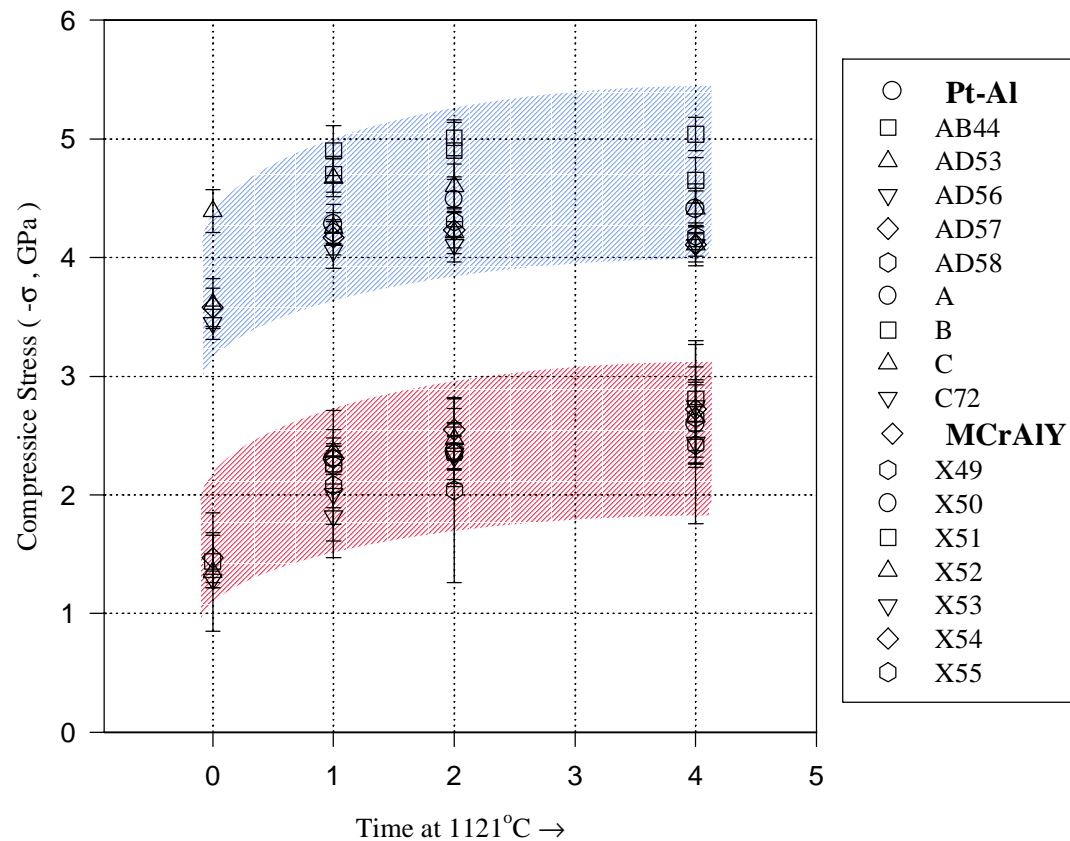
- Measures residual stress in the  $\text{Al}_2\text{O}_3$  layer between the bond coat and the ceramic.

# Laser Fluorescence Results



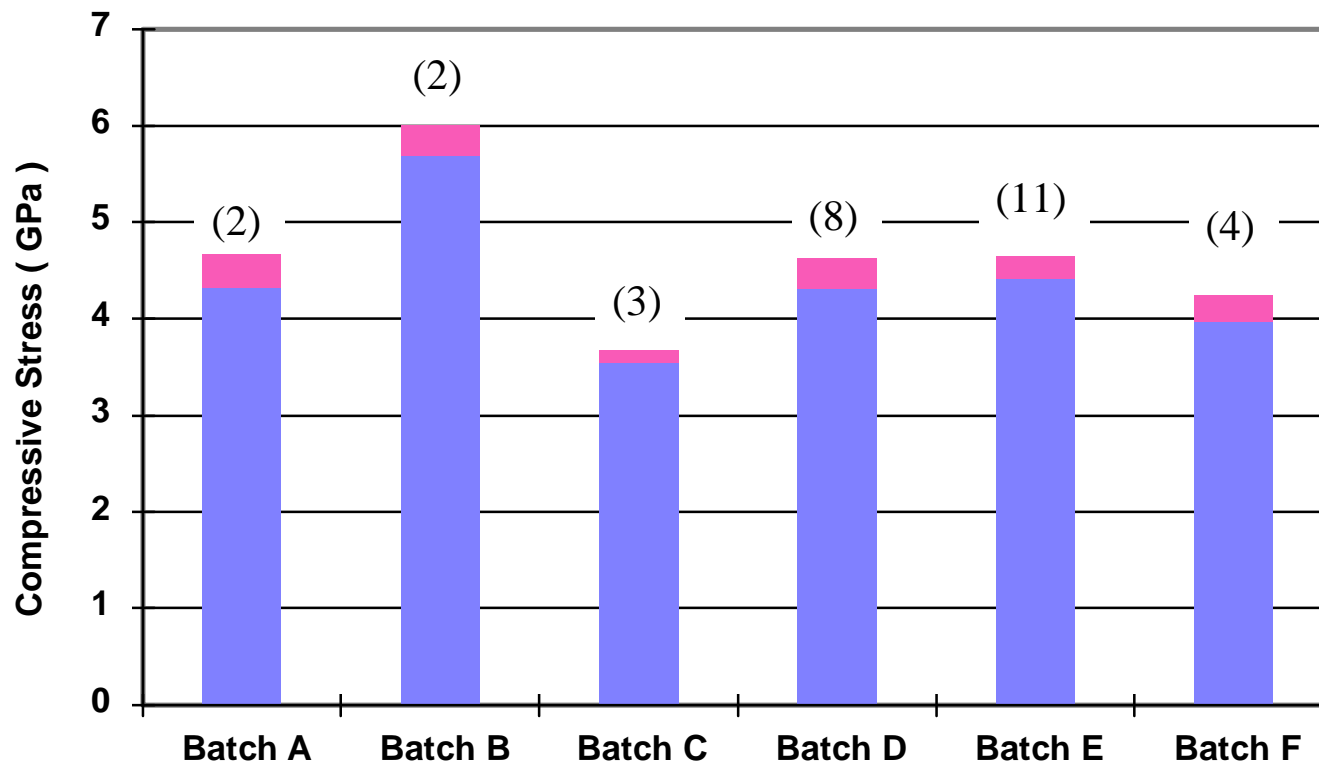
○ Residual stress in the  $\text{Al}_2\text{O}_3$  layer

# Laser Fluorescence Results



○ Residual stress in the  $\text{Al}_2\text{O}_3$  layer vs. time at 1121°C

## Laser Fluorescence Results



- Average compressive stress values of Type I specimens as a function of processing batch.

# Laser Fluorescence Results

